

# Low Temperature Tensile and Fracture Toughness Properties of SCRF Cavity Structural Materials

R. P. Walsh\*, R. R. Mitchell\*\*, V. T. Toplosky\*, R. C. Gentzlinger\*\*

\*National High Magnetic Field Laboratory, Tallahassee, Florida

\*\*Los Alamos National Laboratory, Los Alamos, New Mexico

## Abstract

A materials test program has been performed to support the structural design and analysis of the Accelerator Production of Tritium (APT) cavity assembly. The materials studied here are high purity niobium, commercially pure niobium, commercially pure titanium, and autogenous welds of these three base metals. Tensile tests at 295, 77, and 4 K and fracture toughness tests at 4 K were performed to characterize the mechanical properties of the materials as a function of temperature. The tensile test results are used to evaluate the materials and as input for general design analysis. The fracture toughness test results (some of which represent first time measurements) are used to develop allowable flaw size criteria related to design and fabrication issues. The materials characterization program has been successful in providing key data in areas where the lack of low temperature data prohibited a confident design.

## 1 INTRODUCTION

The lack of available engineering design data for the primary construction materials used for superconducting cavity radio frequency (SCRF) accelerators initiated the materials test program reported here. Niobium, used in SCRF construction because of its' superconducting properties, is also a structural material that is subject to thermal and operating stresses. Materials having body-centered-cubic (BCC) crystal structure such as niobium are known to undergo ductile to brittle transitions making them undesirable for cryogenic applications. Titanium, also used extensively in SCRF construction has a hexagonal-close-packed (HCP) crystal structure and also has brittle tendency at low temperatures. Efficient engineering design requires mechanical properties data at or close to the operating temperature (1.8 K).

Tensile properties can give indications of brittle behavior but do not provide fracture toughness design data. A previous study [1] of high purity niobium (RRR250) tensile properties has shown that the ductility of niobium decreases at 4.2 K. Here two grades of niobium are studied, the high purity grade (RRR250) and a less pure grade (RRR40) along with commercially pure titanium. Tensile and fracture toughness tests are conducted on

niobium, titanium, and autogenous weld versions of the materials, to generate engineering design data.

## 2 MATERIALS

1. High purity niobium (*NbRRR 250*) - 25mm and 4 mm thick hot rolled plate material. The common method for identifying the material is to refer to its' residual resistivity ratio (RRR) number of 250.
2. High purity niobium welded plate (*NbRRR 250 Weld*) - 7 mm thick base metal plates are butt welded using an electron beam weld procedure in a vacuum atmosphere.
3. Commercially pure niobium (*NbRRR 40*)- 19 mm and 9.5 mm thick hot rolled plate material. This material's residual resistivity ratio (RRR) number is 40.
4. Commercially pure niobium welded plate (*NbRRR 40 Weld*) - 7 mm thick base metal plates are butt welded using an electron beam weld procedure in a vacuum atmosphere.
5. Commercially pure titanium (*Ti Grade 2*) - 25mm and 6 mm thick hot rolled plate material.
6. Commercially pure titanium welded plate (*Ti Grade 2 Weld*) - 6 mm thick base metal plates are butt welded using an electron beam weld procedure in a vacuum atmosphere.

## 3 TEST PROCEDURES

Tensile tests are conducted according to guidelines in ASTM E8 and ASTM E1450 standard test methods. The weld material tensile specimens are machined so the tensile axis is transverse to the weld direction. The EB weld region is relatively wide, on the same order as the material thickness and thus occupies about 6 to 7 mm in length of the tensile specimen's 38 mm gage length. Tensile strain is monitored using clip-on extensometers or bonded resistance strain gages. Ambient room temperature accounts for the 295 K test temperature while the 77 K and 4 K temperatures are obtained with boiling liquid nitrogen or liquid helium respectively. Tensile force is applied using displacement control (rate = 0.5 mm/min) resulting in a nominal engineering strain rate of 2e-4 strain sec-1. Elongation measurements are made by scribing the sample with 25 mm gage marks (or 12.7mm for the weld samples) and comparing post-test measurements of the scribe marks. Elongation values for

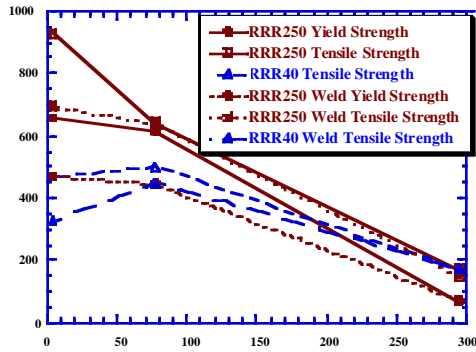


Figure 1: Niobium tensile properties vs temperature.

the welded materials are given for 12.7 mm and 25 mm gage lengths since the weld length is about 6 or 7 mm long.

Fracture toughness tests are conducted according guidelines provided in ASTM E399 and ASTM E813 standard test methods using Compact-Tension (CT) test samples. The sample thickness corresponds to plate thickness. Base metal CT samples are machined in the TL orientation. All precracking of the samples was performed at 77 K. For the weld CT samples, the notch location is centered in the weld width and is oriented parallel to the welding direction. ASTM E399 is used to measure plain strain fracture toughness ( $K_{IC}$ ) which is the linear-elastic fracture criterion. ASTM E813 is the test method used for ductile materials to measure the elastic-plastic fracture criterion ( $J_{IC}$ ).  $J_{IC}$  can be used to obtain an estimate of  $K_{IC}$  but care must be taken when comparing the two values due to assumptions necessary for the comparison. The test matrix is planned such that the base metals are tested using both test methods, E399 ( $K_{test}$ ) for the thick plate and E813 ( $J$  Test) for the thin plate. The weld materials are thin plate, dictated by service conditions and are planned for tests using E813 method.

Table 1: Summary of test results

Material	Yield Strength	Tensile Strength	Fracture Toughness*
	Mpa	Mpa	Mpa*m <sup>0.5</sup>
Nb (RRR250)	654 to 660	924 to 938	55 (+/- 6)
Nb (RRR250) Weld	466 to 473	592 to 779	26 (+/- 3)
Nb (RRR40)	N/A	433 to 525	33 (+/- 7)
Nb (RRR40) Weld	N/A	311 to 341	32 (+/- 3)
Ti Grade 2	926 to 945	1132 to 1165	75 (+/- 11)
Ti Grade 2 Weld	830 to 843	1116 to 1130	90 (+/- 2)**

\* Toughness values are average of a minimum of 3 tests.

\*\* Fracture toughness average of 2 tests.

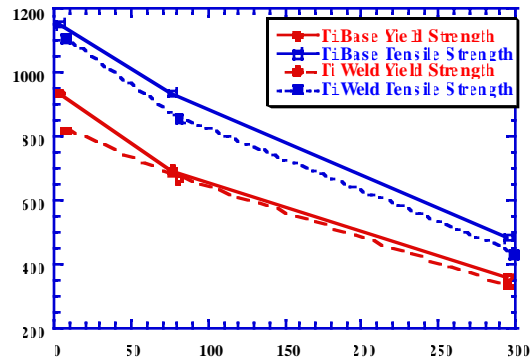


Figure 2: Titanium tensile properties vs temperature.

## 4 RESULTS AND DISCUSSION

The 4 K mechanical properties are summarized in Table 1. These data represent the engineering design values that have been produced in the test program to date. The data presented is based on relatively few tests and is subject to test analysis and interpretation. Further discussion of the test results is given below.

The individual tensile test results are shown in Table 2. The niobium yield and tensile strengths are shown as a function of temperature in Figure 1. The strengths increase dramatically upon cooling from 295 K to 77 K and 4 K. The NbRRR250 and NbRRR40 base materials have equivalent 295 K strength and ductility but the high purity NbRRR250 material has superior low temperature tensile properties. The NbRRR250 retains some ductility (about 15% elongation and 30% reduction of area) at 4 K. The NbRRR40 becomes very brittle at 4 K, having negligible elongation and reduction of area. The NbRRR40 is observed to have little if any plastic deformation in 4 K tensile tests and the material fails before the 0.2% offset yield strength is reached. The decreased yield and tensile strengths of the NbRRR40 at 4 K are attributed to the embrittlement, which in turn must be due to the higher impurity content of the NbRRR40.

The tensile properties of the NbRRR250 weld material are good, at 295 K the strengths are approximately the same as the base metal. Upon cooling, the trend of increasing strength is similar to the NbRRR250 base metal with slightly lower values. The lower strengths at cryogenic temperatures should be expected since welding increases the grain size and may introduce impurities or cause existing impurities to migrate to grain boundaries. The NbRRR40 weld strength is approximately the same as the NbRRR40 base metal at 295 K. Upon cooling, the trend is similar to the NbRRR40 base metal with slightly lower values. The 4 K embrittlement of the NbRRR40 is amplified for the welded condition. The elongation and reduction of area of the NbRRR40 at 77 K and 4 K are negligible.

Table 2: Tensile test results.

Mat'l	Temp	Yield Stress	Tensile Strength	Red. in Area	Elong.- 12.7 mm	Elong.- 25 mm
	K	Mpa	Mpa	%	%	%
Nb	295	67	172	88.3		57.4
RRR	77	618	642	72.4		30.5
250	4	658	929	28.7		16.0
RRR	295	70	151	86.7		28.2
250	77	445	639	59.6		13.4
WELD	4	470	696	10.4	9.1	4.2
Nb	295	76	171	80.9		41.05
RRR	77	443	502	7.3		3.1
40	4		468	1.1		1.6
RRR	295	95.5	172	90.0		58.5
40	77		450	0.2		1.4
WELD	4		327	0.4		1.36
Ti	295	358	481	60.5		32.6
Gr. 2	77	695	936	55.7		50.9
	4	936	1153	34.3		35.6
Ti	295	332	446	63.0		14.3
Gr 2	77	681	872	48.0		33.8
WELD	4	834	1123	32.3		27.6

The tensile properties of the Ti Grade 2 are also shown in Tables 1 and 2. The tensile properties of the both the base metal and the weld are excellent. The increase in strength as temperature decreases, shown in Figure 2, is as expected. The Ti Grade 2 weld material behaves as expected with strengths slightly lower than the base metal along the same trend lines.

The fracture toughness values presented in Table 1 are qualitative and may not represent the actual plain-strain fracture toughness of these materials. Fracture toughness testing at 4 K presents difficulties in meeting validity requirements of the fracture toughness test standards. The niobium materials behaved too brittle for J tests but had just enough ductility to invalidate the plain strain requirements of the K test. The titanium fracture toughness tests results are more reliable but there were subtle problems with meeting all validity requirements.

In general, the measured 4 K fracture toughness of the niobium materials is very low and pursuit of a perfect test to measure how low it really is has diminishing returns. The NbRRR250 base metals are tested using ASTM E399 for the thick (25mm) sample and ASTM E813 for the 4 mm thick material as planned. The results from the two materials and methods agree relatively well. The NbRRR250 base metal has the highest average 4 K toughness ( $55 \text{ Mpa}\cdot\text{m}^{0.5}$ ) of the four niobium conditions tested. The NbRRR40 base metals are also tested using ASTM E399 for the thick (19mm) sample and ASTM E813 for the thin (9.5 mm) material but the 9.5 mm thick material is too brittle for J tests and must be tested using

the K test (E399) method. The average estimated toughness of the NbRRR40 material is  $33 \text{ Mpa}\cdot\text{m}^{0.5}$ . The average estimated toughness of the NbRRR250 weld and NbRRR40 weld are  $26 \text{ Mpa}\cdot\text{m}^{0.5}$  and  $32 \text{ Mpa}\cdot\text{m}^{0.5}$  respectively.

The average 4 K fracture toughness of the Ti Grade 2 base metal and weld metal are  $75 \text{ Mpa}\cdot\text{m}^{0.5}$  and  $90 \text{ Mpa}\cdot\text{m}^{0.5}$  respectively. The tougher weld metal was unexpected, and may not be accurate due to an insufficient number of tests and problems with crack front curvature. Residual stress in the welded titanium plate caused the fatigue crack front to be slanted, as samples consistently have pre-cracks that are longer on one side of the plate than the other. The 25 mm and 6 mm thick titanium base metals had sufficient ductility to be J tested (ASTM E813). The data for the weld represents one J test and one K test.

## 5 CONCLUSIONS

The materials characterization program has been successful in providing key data in areas where the lack of low temperature data prohibited a confident design. The data presented is based on relatively few tests; the fracture toughness values presented are qualitative and represent estimates of the plain-strain fracture toughness of these materials.

The comparison of two purity grades of niobium shows there is a large effect of the purity on the low temperature properties. The lower purity niobium (NbRRR40) exhibits extreme brittleness in 4 K tensile tests, failing at a stress about one-half that of the NbRRR250. The most conservative design data with respect to the fracture toughness of niobium at 4 K is the minimum value ( $23 \text{ Mpa}\cdot\text{m}^{0.5}$ ) observed for the NbRRR250welded condition.

The Ti Grade 2 shows good tensile strength and ductility at 4 K. The minimum fracture toughness data attained for the titanium is  $64 \text{ Mpa}\cdot\text{m}^{0.5}$  which is observed for the base material rather than the weld.

## 6 REFERENCES

- [1] Rao, M.G. and Kneisel, P., Mechanical Properties of High RRR Niobium at Cryogenic Temperatures, Adv. In Cryo. Engr., Vol. 40, pp.1383-1390